

WHAT IS CLAIMED IS:

1           1.       An apparatus for parallel processing of reaction mixtures comprising:  
2           vessels for containing the reaction mixtures;  
3           a stirring system for agitating the reaction mixtures; and  
4           a temperature control system that is adapted to maintain a first group of the vessels at  
5           a different temperature than a second group of the vessels.

1           2.       The apparatus of claim 1, further comprising a reactor block;  
2           wherein the vessels comprise wells formed in the reactor block.

1           3.       The apparatus of claim 2, wherein the vessels further comprise removable  
2           liners, each of the liners having an interior surface defining a cavity for containing one of the  
3           reaction mixtures and an exterior surface dimensioned so that the liners fit within the wells  
4           formed in the reactor block.

1           4.       The apparatus of claim 3, wherein the removable liners are glass vials.

1           5.       The apparatus of claim 3, further comprising an insulating material filling gaps  
2           between the removable liners and the wells.

1           6.       The apparatus of claim 5, wherein the insulating material is glass wool or  
2           silicone rubber.

1           7.       The apparatus of claim 3, further comprising a conductive material filling gaps  
2           between the removable liners and the wells.

1           8.       The apparatus of claim 7, wherein the conductive material is a thermal paste.

1           9.       The apparatus of claim 2, wherein the wells comprise holes extending from a  
2           top surface of the reactor block to a bottom surface of the reactor block, the apparatus further  
3           comprising:

4           a removable lower plate disposed on the bottom surface of the reactor block, the  
5           removable lower plate defining a base of each of the wells; and

6           a removable upper plate disposed on the top surface of the reactor block, the  
7           removable upper plate defining an upper end of each of the wells.

1           10.    The apparatus of claim 9, wherein the removable upper plate further comprises  
2   vessel seals in substantial alignment with the wells, the vessel seals allowing processing of  
3   the reaction mixtures at pressures different than atmospheric pressure.

1           11.    The apparatus of claim 2, further comprising a passageway formed in the  
2   reactor block adapted to provide a flow path for a thermal fluid and adapted to provide heat  
3   exchange between the vessels and the thermal fluid.

1           12.    The apparatus of claim 2, further comprising:  
2           a passageway formed in the reactor block adapted to provide a flow path for a thermal  
3   fluid and to provide heat exchange between the thermal fluid and the vessels.

1           13.    The apparatus of claim 2, further comprising:  
2           thermoelectric devices and a heat transfer plate, the thermoelectric devices disposed  
3   between the reactor block and the heat transfer plate such that the thermoelectric devices  
  transfer heat from the vessels to the heat transfer plate or from the heat transfer plate to the  
  vessels.

1           14.    The apparatus of claim 13, wherein each of the thermoelectric devices  
2   transfers heat predominantly to or from a single vessel.

1           15.    The apparatus of claim 13, wherein the heat transfer plate further comprises a  
2   passageway formed in the heat transfer plate adapted to provide a flow path for a thermal  
3   fluid.

1           16.    The apparatus of claim 1, further comprising a chamber enclosing the vessels,  
2   the chamber being substantially gas impermeable.

1           17.    The apparatus of claim 1, further comprising a plurality of modules, each of  
2   the modules containing a portion of the vessels.

1           18.    The apparatus of claim 17, wherein each of the modules comprises a block and  
2   wherein vessels comprise wells formed in the block.

1           19.    The apparatus of claim 1, further comprising a robotic material handling  
2   system for loading the vessels with starting materials.

1        20.    The apparatus of claim 1, wherein the temperature control system includes a  
2    temperature monitoring system comprising:  
3        temperature sensors in thermal contact with the vessels; and  
4        a temperature monitor that communicates with the temperature sensors and converts  
5    signals received from the temperature sensors to a standard temperature scale.

1        21.    The apparatus of claim 20, wherein the temperature sensors are  
2    thermocouples, resistance thermometric devices, or thermistors, alone or in combination.

1        22.    The apparatus of claim 20, further comprising a processor that communicates  
2    with the temperature monitor, the processor adapted to perform calculations on data received  
3    from the temperature monitor.

1        23.    The apparatus of claim 1, wherein the temperature control system includes a  
2    remote temperature monitoring system comprising an infrared-sensitive camera positioned to  
3    detect infrared radiation emanating from each of the vessels.

1        24.    The apparatus of claim 23, wherein each of the vessels are fitted with a cap  
2    that transmits infrared radiation.

1        25.    The apparatus of claim 23, further comprising an isolation chamber enclosing  
2    the vessels.

1        26.    The apparatus of claim 1, wherein the temperature control system further  
2    comprises temperature sensors and heat transfer devices, the temperature sensors and the heat  
3    transfer devices in thermal contact with the vessels, and the heat transfer devices adapted to  
4    transfer heat to or from the vessels in response to signals received from the temperature  
5    sensors.

1        27.    The apparatus of claim 26, wherein the temperature control system further  
2    comprises:

3        a processor that communicates with the temperature sensors and the heat transfer  
4    devices, the processor and the heat transfer devices adapted to adjust heat flow to or from the

5 heat transfer devices in response to signals received by the processor from the temperature  
6 sensors.

1 28. The apparatus of claim 26, wherein the heat transfer devices are electric  
2 resistance heaters.

1 29. The apparatus of claim 28, wherein the temperature sensors and the electric  
2 resistance heaters are the same.

1 30. The apparatus of claim 29, wherein the temperature sensors and the electric  
2 resistance heaters are thermistors.

1 31. The apparatus of claim 26, wherein the heat transfer devices are thermoelectric  
2 devices.

1 32. The apparatus of claim 26, wherein the temperature control system further  
2 comprises a uniform temperature reservoir in thermal contact with the vessels.

1 33. The apparatus of claim 32, wherein the uniform temperature reservoir is  
2 adapted to contain a thermal fluid.

1 34. The apparatus of claim 33, wherein the vessels are suspended in the uniform  
2 temperature reservoir.

1 35. The apparatus of claim 32, wherein the temperature control system further  
2 comprises a heat pump in thermal contact with the uniform temperature reservoir, the heat  
3 pump adapted to maintain the uniform temperature reservoir at a selected temperature.

1 36. The apparatus of claim 1, wherein the stirring system comprises:  
2 stirring members at least partially contained in the vessels; and  
3 a drive mechanism coupled to the stirring members, the drive mechanism adapted to  
4 rotate the stirring members.

1 37. The apparatus of claim 36, wherein the drive mechanism comprises motors  
2 coupled to the stirring members so that the speed, torque, or speed and torque of the stirring

1 38. The apparatus of claim 37, further comprising strain gauges for measuring  
2 torque exerted by the motors on the reaction mixtures, wherein the motors are rigidly attached  
3 to a motor support, and the strain gauges are mounted between the motor support and the  
4 motors.

1 39. The apparatus of claim 37, further comprising speed sensors integral to the  
2 motors for monitoring rotational speed of the stirring members.

1 40. The apparatus of claim 39, further comprising a processor in communication  
2 with the speed sensors, the processor adjusting power supplied to each of the motors in  
3 response to signals received from the speed sensors to maintain the rotational speed of the  
4 stirring members at selected values.

1 41. The apparatus of claim 36, further comprising strain gauges for measuring  
2 torque exerted by the drive mechanism on the reaction mixtures, each of the strain gauges  
3 having a first end and a second end, the first end of each of the strain gauges rigidly attached  
4 to the vessel support, and the second end of each of the strain gauges rigidly attached to the  
5 vessels such that each of the vessels is attached to one of the strain gauges.

1 42. The apparatus of claim 41, further comprising:  
2 first permanent magnets attached to the second end of each of the strain gauges; and  
3 second permanent magnets attached to the vessels so that magnetic coupling between  
4 the first permanent magnets and the second permanent magnets prevents the vessels from  
5 rotating.

1 43. The apparatus of claim 36, wherein the drive mechanism comprises:  
2 a motor; and  
3 a drive train coupling the motor to the stirring members.

1 44. The apparatus of claim 43, wherein the drive train comprises:  
2 gears attached to the motor and to portions of the stirring members located external to  
3 the vessels, each of the gears dimensioned and arranged to mesh with at least one adjacent  
4 gear so that rotational energy is transmitted along the drive train from the motor to the stirring  
5 members.

1 45. The apparatus of claim 36, wherein each of the stirring members comprises:  
2 a spindle, each spindle having a first end and a second end; and  
3 a stirring blade attached to the first end of the spindle.

1 46. The apparatus of claim 45, wherein the second end of the spindle is  
2 mechanically coupled to the drive mechanism.

1 47. The apparatus of claim 45, wherein the second end of the spindle is  
2 magnetically coupled to the drive mechanism.

1 48. The apparatus of claim 45, further comprising a strain gauge located within the  
2 spindle.

1 49. The apparatus of claim 45, further comprising an optical speed sensor mounted  
2 adjacent to the spindle for monitoring rotational speed of the spindle.

1 50. The apparatus of claim 36, wherein the stirring members are magnetic stirring  
2 bars, and the drive mechanism comprises an array of electromagnets that produce rotating  
3 magnetic fields in the vessels.

1 51. The apparatus of claim 50, wherein the array of electromagnets is arranged so  
2 that each of the vessels is located between four electromagnets, the four electromagnets  
3 defining four corners of a quadrilateral sub-array.

1 52. The apparatus of claim 50, wherein the array of electromagnets comprises:  
2 a first group of electromagnets, and a second group of electromagnets, the first group  
3 of electromagnets electrically connected in series so that pairs of successive electromagnets  
4 define two opposite corners of each quadrilateral sub-array, and the second group of  
5 electromagnets electrically connected in series such that pairs of successive electromagnets  
6 define two opposite corners of each quadrilateral sub-array.

1 53. The apparatus of claim 52, further comprising a drive circuit and a processor,  
2 the drive circuit controlled by the processor and adapted to independently and temporally  
3 vary electrical current in the first group of electromagnets and in the second group of  
4 electromagnets.

1 54. The apparatus of claim 53, wherein the drive circuit further comprises a power  
2 source that is adapted to provide sinusoidal electrical currents.

1 55. The apparatus of claim 53, wherein the drive circuit further comprises a power  
2 source that is adapted to provide pulsed electrical currents.

1 56. The apparatus of claim 50, wherein the array of electromagnets is mounted on  
2 a printed circuit board.

1 57. The apparatus of claim 36, wherein the stirring system further comprises a  
2 system for measuring phase lag between the stirring members and the drive mechanism.

1 58. The apparatus of claim 57, wherein the system for measuring phase lag  
2 comprises inductive sensing coils located adjacent to the vessels and displaced laterally from  
3 the axes of rotation of the stirring members, and a phase-sensitive detector adapted to monitor  
4 phase differences among signals generated by the inductive sensing coils and a reference  
5 signal having the same phase behavior as the drive mechanism.

1 59. The apparatus of claim 1, further including a system for evaluating reaction  
2 mixtures comprising at least one mechanical oscillator in contact with the reaction mixtures,  
3 the at least one mechanical oscillator adapted to receive a variable frequency excitation signal  
4 and to transmit a response signal that depends on one or more material properties of the  
5 reaction mixtures.

1 60. The apparatus of claim 59, further comprising a plurality of mechanical  
2 oscillators, each of the mechanical oscillators located in the vessels.

1 61. The apparatus of claim 59, wherein the at least one mechanical oscillator is a  
2 tuning fork resonator or bimorph/unimorph resonator.

1 62. The apparatus of claim 59, further comprising a three-axis translation system  
2 for placing the at least one mechanical oscillator in the vessels.

1 63. The apparatus of claim 59, further comprising:  
2 a probe adapted to withdraw and dispense the reaction mixtures; and

3 a three-axis translation system coupled to the probe for manipulating the probe  
4 position;

5 wherein the at least one mechanical oscillator is located in a separate vessel and the  
6 three-axis translation system is adapted to withdraw a portion of one of the reaction mixtures  
7 from one of the vessels and to dispense the portion of one of the reaction mixtures in the  
8 separate vessel.

1 64. The apparatus of claim 1, further comprising a pressure control system.

1 65. The apparatus of claim 64, wherein each of the vessels has a gas inlet for  
2 introducing a vapor-phase component of the reaction mixtures into the vessels.

1 66. The apparatus of claim 65, further comprising a flow sensor located between  
2 the gas inlet and a source of the vapor-phase component of the reaction mixture.

2 67. The apparatus of claim 64, wherein the vessels have a removable seal for  
loading the vessels with condensed-phase components of the reaction mixtures.

2 68. The apparatus of claim 64, further comprising temperature sensors in thermal  
contact with the vessels, the temperature sensors providing data for determining pressure  
3 corrections based on temperature changes of the reaction mixtures.

2 69. An apparatus for monitoring rates of production or consumption of a gas-  
phase component of a reaction mixture comprising:

3 a vessel for containing the reaction mixture, the vessel having a gas inlet for  
4 introducing a gas-phase component of the reaction mixture into the vessel and a removable  
5 seal for loading the vessel with one or more condensed-phase components of the reaction  
6 mixture;

7 a temperature control system for regulating the temperature of the reaction mixture;  
8 and

9 a pressure control system comprising:

10 a pressure sensor in fluid communication with the vessel;

11 a conduit providing fluid communication between a source of the gas-phase  
12 component and the gas inlet of the vessel;



13 a valve located along the conduit between the source of the gas-phase component and  
14 the gas inlet of the vessel;  
15 a valve controller communicating with the valve, the valve controller regulating the  
16 amount of the gas-phase component entering the vessel by selectively opening or closing the  
17 valve; and  
18 a processor communicating with the valve controller and the pressure sensor, the  
19 processor directing the valve controller to selectively open or close the valve in response to a  
20 signal received from the pressure sensor.

1 70. The apparatus of claim 69, further comprising a temperature sensor in thermal  
2 contact with the vessel, the temperature sensor communicating with the processor and  
3 providing the processor with data for determining pressure corrections based on temperature  
4 changes of the reaction mixture.

2 71. The apparatus of claim 69, further comprising a flow sensor located along the  
3 conduit between the valve and the gas inlet of the vessel, the flow sensor communicating with  
4 the processor and providing the processor with data for determining an amount of the gas-  
5 phase component entering the vessel.

2 72 An apparatus for monitoring rates of consumption of a gas-phase reactant of a  
3 reaction mixture comprising:

3 a vessel for containing the reaction mixture, the vessel having a gas inlet for  
4 introducing the gas-phase reactant into the vessels and a removable seal for loading the vessel  
5 with one or more condensed-phase components of the reaction mixture;

6 a temperature control system for regulating the temperature of the reaction mixture;  
7 and

8 a pressure control system comprising:

9 a pressure sensor in fluid communication with the vessel;

10 a conduit providing fluid communication between a source of the gas-phase reactant  
11 and the gas inlet of each of the vessels;

12 a flow sensor located along the conduit between the source of the gas-phase  
13 component and the gas inlet of the vessel; and

14 a processor communicating with the flow sensor, the flow sensor providing the  
15 processor with data for determining an amount of the gas-phase reactant entering the vessel  
16 during processing.

1 73. A method of making and characterizing materials comprising the steps of:  
2 providing vessels with starting materials to form reaction mixtures;  
3 allowing the reaction mixtures in the vessels to react;  
4 stirring the reaction mixtures during at least a portion of the reaction; and  
5 evaluating the reaction mixtures by measuring at least one characteristic of the  
6 reaction mixtures during at least a portion of the reaction;  
7 wherein the reaction is carried out at about the same time for each of the reaction  
8 mixtures.

1 74. The method of claim 73, wherein the vessels are provided with starting  
2 materials using a robotic material.

1 75. The method of claim 73, further comprising blanketing the vessels in an inert  
2 gas atmosphere while providing vessels with starting materials.

1 76. The method of claim 73, wherein the reaction mixtures are evaluated by  
2 monitoring temperatures of each of the reaction mixtures.

1 77. The method of claim 76, wherein monitoring temperatures comprises detecting  
2 infrared emissions from the reaction mixtures.

1 78. The method of claim 73, wherein the reaction mixtures are evaluated by  
2 monitoring heat transfer rates into or out of the vessels.

1 79. The method of claim 78, wherein monitoring heat transfer rates comprises:  
2 measuring temperature differences between each of the reaction mixtures and a  
3 thermal reservoir surrounding the vessels; and  
4 determining heat transfer rates from a calibration relating the temperature differences  
5 to heat transfer rates.

1 80. The method of claim 78, further comprising computing conversion of the  
2 starting materials based on the heat transfer rates of the monitoring step.

1 81. The method of claim 80, further comprising determining rates of reaction  
2 based on conversion of the starting materials.

1 82. The method of claim 73, wherein stirring comprises:  
2 supplying the reaction mixtures with stirring members; and  
3 rotating each of the stirring members.

1 83. The method of claim 82, wherein the stirring members rotate at the same rate.

1 84. The method of claim 82, wherein the reaction mixtures are evaluated by  
2 monitoring the torque needed to rotate the stirring members.

1 85. The method of claim 84, wherein the torque is monitored by measuring phase  
2 lag between the torque and the stirring members.

1 86. The method of claim 84, wherein the reaction mixtures are evaluated by  
2 determining viscosity of each of the reaction mixtures from a calibration relating the torque  
3 and viscosity.

1 87. The method of claim 86, wherein the reaction mixtures are evaluated by:  
2 measuring heat transfer rates into or out of the vessels;  
3 computing conversion of the starting materials based on heat transfer rates into or out  
4 of the vessels; and  
5 calculating molecular weight of a component of the reaction mixtures based on  
6 conversion of the starting materials and on viscosity of each of the reaction mixtures.

1 88. The method of claim 82, wherein the evaluating step further comprises the  
2 step of monitoring the power needed to rotate each of the stirring members in the rotating  
3 step.

1 89. The method of claim 88, wherein the reaction mixtures are evaluated by  
2 determining viscosity of each of the reaction mixtures from a calibration relating power and  
3 viscosity.

1 90. The method of claim 89, wherein the reaction mixtures are evaluated by:  
2 measuring heat transfer rates into or out of the vessels;  
3 computing conversion of the starting materials based on heat transfer into or out of the  
4 vessels; and  
5 calculating molecular weight of a component of the reaction mixtures based on  
6 conversion of the starting materials and on viscosity of each of the reaction mixtures.

1 91. The method of claim 82, wherein the reaction mixtures are evaluated by  
2 monitoring stall frequencies of the stirring members in the rotating step.

1 92. The method of claim 91, wherein the reaction mixtures are evaluated by  
2 determining viscosity of each of the reaction mixtures from a calibration relating stall  
3 frequencies and viscosity.

1 93. The method of claim 92, wherein the reaction mixtures are evaluated by:  
2 measuring rates of heat transfer into or out of the vessels;  
3 computing conversion of the starting materials based on rates of heat transfer into or  
4 out of the vessels; and  
5 calculating molecular weight of a component of the reaction mixtures based on  
6 conversion of the starting materials and on viscosity of each of the reaction mixtures.

1 94. The method of claim 73, wherein the reaction mixtures are evaluated by:  
2 stimulating mechanical oscillators in contact with the reaction mixtures with a  
3 variable frequency excitation signal;  
4 monitoring response signals from the mechanical oscillators;  
5 determining a property of each of the reaction mixtures from a calibration relating  
6 response signals and the property.

1 95. The method of claim 94, wherein the property is molecular weight, specific  
2 gravity, elasticity, dielectric constant or conductivity.

1 96. The method of claim 73, wherein the reaction mixtures are evaluated by:  
2 supplying a separate vessel with a mechanical oscillator;  
3 placing a portion of a particular reaction mixture in the separate vessel;  
4 stimulating the oscillator with a variable frequency excitation signal;  
5 monitoring a response signal from the mechanical oscillator; and  
6 determining a property of the particular reaction mixture from a calibration relating  
7 response signals and the property.

1 97. The method of claim 73, wherein there is a net loss of moles of gas-phase  
2 components of each of the reaction mixtures due to reaction, and the reaction mixtures are  
3 evaluated by:

4 filling the vessels with a gas-phase reactant until gas pressure in each of the vessels  
5 exceeds an upper-pressure limit,  $P_U$ ;  
6 allowing gas pressure in each of the vessels to decay below a lower-pressure limit,  $P_L$ ;  
7 monitoring the gas pressure in each of the vessels to generate a record of pressure  
8 versus time for each of the vessels;  
9 repeating filling, allowing, and monitoring at least once; and  
10 determining rates of consumption of the gas-phase reactant in each of the reaction  
11 mixtures from the record of pressure versus time for each of the vessels.

1 98. The method of claim 97, wherein the rates of consumption are determined by:  
2 converting the record of pressure versus time for each of the vessels to partial pressure  
3 of the gas-phase reactant versus time for each of the vessels; and  
4 calculating rates of consumption of the gas-phase reactant in each of the reaction  
5 mixtures from time rates of change of the partial pressure of the gas-phase reactant versus  
6 time during the gas pressure decays.

1 99. The method of claim 97, wherein the rates of consumption are determined  
2 from frequencies of the filling steps over particular time intervals.

1 100. The method of claim 97, wherein the rates of consumption are determined by:  
2 estimating a volumetric flow rate of the gas-phase reactant entering a given vessel

4 multiplying the volumetric flow rate of the gas-phase reactant entering the given  
5 vessel during the particular filling operation by an amount of time elapsed during the  
6 particular filling operation to obtain an estimate of an amount of the gas-phase reactant  
7 entering the given vessel during the particular filling operation;

8 dividing the estimate of the amount of the gas-phase reactant entering the given vessel  
9 during the particular filling step by an amount of time elapsed during a subsequent gas  
10 pressure decay to obtain an average rate of consumption of the gas-phase reactant in the given  
11 vessel for the particular filling operation and the subsequent gas pressure decay.

1 101. The method of claim 97, wherein the rates of consumption are determined by:  
2 measuring an amount of the gas-phase reactant entering a given vessel during a  
3 particular filling operation;

4 dividing the amount of the gas-phase reactant entering the given vessel during the  
5 particular filling operation by an amount of time elapsed during a subsequent gas pressure  
6 decay to obtain an average rate of consumption of the gas-phase reactant in the given vessel  
7 for the particular filling operation and the subsequent gas pressure decay.

8 102. The method of claim 73, wherein there is a net gain of moles of gas-phase  
9 components of each of the reaction mixtures due to reaction, and the reaction mixtures are  
10 evaluated by:

11 allowing gas pressure in each of the vessels to rise above an upper-pressure limit,  $P_H$ ;  
5 venting the vessels until gas pressure in each of the vessels falls below a lower-  
6 pressure limit,  $P_L$ ;

7 monitoring the gas pressure in each of the vessels during the gas pressure rise and  
8 venting to generate a record of pressure versus time for each of the vessels;

9 repeating allowing, venting, and monitoring at least once; and

10 determining rates of production of a gas-phase product in each of the reaction  
11 mixtures from the record of pressure versus time for each of the vessels.

1 103. The method of claim 102, wherein the rates of production are determined by:  
2 converting the record of pressure versus time for each of the vessels to partial pressure  
3 of the gas phase product versus time for each of the vessels;

calculating rates of production of the gas-phase product in each of the reaction mixtures from time rates of change of the partial pressure of the gas-phase product versus time during the gas pressure rises.

104. The method of claim 102, wherein the rates of production are determined from frequencies of venting over particular time intervals.

105. The method of claim 102, wherein the rates of production are determined by: estimating a volumetric flow rate of the gas-phase product leaving a given vessel during a particular venting operation; multiplying the volumetric flow rate of the gas-phase product leaving the given vessel during the particular venting operation by an amount of time elapsed during the particular venting operation to obtain an estimate of an amount of the gas-phase product leaving the given vessel during the particular venting operation;

dividing the estimate of the amount of the gas-phase product leaving the given vessel during the particular venting operation by an amount of time elapsed during a subsequent gas pressure rise to obtain an average rate of production of the gas-phase product in the given vessel for the particular venting operation and the subsequent gas pressure rise.

106. The method of claim 102, wherein the rates of production are determined by: measuring an amount of the gas-phase product leaving a given vessel during a particular venting operation;

dividing the amount of the gas-phase product leaving the given vessel during the particular venting operation by an amount of time elapsed during a subsequent gas pressure rise to obtain an average rate of production of the gas-phase product in the given vessel for the particular venting operation and the subsequent gas pressure rise.

107. The method of claim 73, further comprising controlling temperatures of each of the reaction mixtures.

108. The method of claim 107, wherein temperatures of each of the reaction mixtures are controlled independently.

109. An apparatus for parallel processing of reaction mixtures comprising: vessels for containing the reaction mixtures;

3 a stirring system for agitating the reaction mixtures;  
4 a temperature control system for regulating the temperature of the reaction mixtures;  
5 and  
6 an injection system for introducing a fluid into the vessels at a pressure different than  
7 ambient pressure.

1 110. The apparatus of claim 109, wherein the injection system comprises:  
2 fill ports adapted to receive a fluid delivery probe;  
3 first conduits and valves, the first conduits providing fluid communication between  
4 the fill ports and the valves; and  
5 second conduits and injectors, the second conduits providing fluid communication  
6 between the valves and the injectors;  
7 wherein the injectors are located in the vessels.

1 111. The apparatus of claim 110, further comprising a robotic fluid handling  
2 system, wherein the robotic fluid handling system is adapted to manipulate the fluid delivery  
3 probe.

1 112. The apparatus of claim 111, further comprising a computer to control both the  
2 robotic fluid handling system and the valves.

1 113. The apparatus of claim 110, wherein the fill port comprises:  
2 an elongated body having a longitudinal axis and a bore centered on the longitudinal  
3 axis, the bore extending the length of the elongated body and characterized by first, second,  
4 and third diameters, wherein the first diameter is greater than the second diameter, and the  
5 second diameter is greater than the third diameter;

6 an elastomeric o-ring seated within the bore of the elongated body on a first ledge  
7 defined by the second diameter and the third diameter; and

8 a cylindrical sleeve having a hole centered on its axis of rotation, the hole extending  
9 the length of the cylindrical sleeve;

10 wherein the cylindrical sleeve is seated within the bore of the elongated body on a  
11 second ledge defined by the first diameter and the second diameter and the cylindrical sleeve  
12 abuts the elastomeric o-ring.



1 114. The apparatus of claim 113, wherein the cylindrical sleeve is made of a  
2 chemically resistant plastic material.

1 115. The apparatus of claim 114, wherein the chemically resistant plastic material is a  
2 perfluoro-elastomer or polyethylethylketone or polytetrafluoroethylene.

1 116. The apparatus of claim 110, wherein the fill port comprises:  
2 an elongated body having a longitudinal axis and a bore centered on the longitudinal  
3 axis, the bore extending the length of the elongated body and characterized by a first diameter  
4 and a second diameter, wherein the first diameter is greater than the second diameter; and  
5 a cylindrical insert having a tapered hole centered on its axis of rotation, the tapered  
6 hole extending the length of the cylindrical insert;  
7 wherein the cylindrical insert is seated within the bore of the elongated body on a  
8 ledge defined by the first diameter and the second diameter.

1 117. The apparatus of claim 116, wherein the cylindrical insert is made of a  
2 chemically resistant plastic material.

1 118. The apparatus of claim 117, wherein the chemically resistant plastic material is  
2 a perfluoro-elastomer or polyethylethylketone or polytetrafluoroethylene.

1 119. The apparatus of claim 110, further comprising a reactor block;  
2 wherein the vessels comprise wells formed in the reactor block.

1 120. The apparatus of claim 119, further comprising an injector manifold associated  
2 with the reactor block;  
3 wherein the fill ports and valves are in fluid communication with the injector  
4 manifold.

1 121. The apparatus of claim 120, wherein the injector manifold is attached to the  
2 reactor block, and the first conduits and the second conduits are passageways formed in the  
3 injector manifold.

122. The apparatus of claim 120, wherein the wells comprise holes extending from a top surface of the reactor block to a bottom surface of the reactor block, the apparatus further comprising:

a lower plate disposed on the bottom surface of the reactor block, the lower plate defining a base of each of the wells;

an injector adapter plate disposed on the top surface of the reactor block, the injector adapter plate having holes substantially aligned with the wells and having channels extending from a front edge of the injector adapter plate to a bottom surface of the injector adapter plate, wherein the injectors are attached to the bottom surface of the injector adapter plate and are in fluid communication with the channels, and the injector manifold is attached to the front edge of the injector adapter plate so that the second conduits are in fluid communication with the channels of the injector adapter plate; and

an upper plate disposed on the injector adapter plate, the upper plate defining an upper end of each of the well

123. The apparatus of claim 122, wherein the injectors extend into the reaction mixtures.

124. An apparatus for parallel processing of reaction mixtures comprising: vessels for containing the reaction mixtures, the vessels sealed to minimize unintentional gas flow into or out of the vessels;

a temperature control system for regulating the temperature of the reaction mixtures; and

a stirring system for agitating the reaction mixtures, the stirring system comprising: spindles contained in the vessels, each of the spindles having a first end and a second end;

a stirring blade attached to the first end of each of the spindles;

a drive mechanism located external to the vessels that is adapted to rotate the spindles; and

magnetic feed through devices for magnetically coupling the drive mechanism to the second end of each of the spindles.

1 125. The apparatus of claim 124, wherein each of the magnetic feed through  
2 devices comprises:

3 a rigid cylindrical pressure barrier having an interior surface that together with one of  
4 the vessels defines a closed chamber;

5 a magnetic driver rotatably mounted concentrically with the pressure barrier and  
6 external to the closed chamber; and

7 a magnetic follower rotatably mounted within the closed chamber;

8 wherein the drive mechanism is mechanically coupled to the magnetic driver and the  
9 magnetic follower follows the magnetic driver, and the second end of one of the spindles is  
10 attached to the magnetic follower so that the spindles rotate as driven by the drive  
11 mechanism.

1 126. The apparatus of claim 125, wherein the drive mechanism further comprises:

2 a motor; and

3 a drive train coupling the motor to the magnetic driver of the magnetic feed through  
4 devices.

1 127. The apparatus of claim 125, wherein the drive train comprises:

2 gears attached to the motor and to the magnetic driver of the magnetic feed through  
3 devices, each of the gears dimensioned and arranged so as to mesh with at least one adjacent  
4 gear so that rotational energy is transmitted along the drive train from the motor to the  
5 spindles through the magnetic feed through devices.

1 128. An apparatus for parallel processing of reaction mixtures comprising:

2 vessels for containing the reaction mixtures;

3 a temperature control system for regulating the temperature of the reaction mixtures;

4 and

5 a stirring system for agitating the reaction mixtures, the stirring system comprising  
6 multi-piece spindles partially contained in the vessels, and a drive mechanism coupled to the

1 129. The apparatus of claim 128, wherein the removable stirrer is made of a  
2 chemically resistant plastic material.

1 130. The apparatus of claim 129, wherein the chemically resistant plastic material is  
2 a perfluoro-elastomer or polyethylethylketone or polytetrafluoroethylene or glass.

1 131. The apparatus of claim 129, further comprising a coupler for reversibly  
2 attaching the removable stirrer to the upper spindle portion, the coupler comprising:  
3 a cylindrical body having first and second holes centered along an axis of rotation of  
4 the coupler, the first hole dimensioned to receive an end of the upper spindle portion, and the  
5 second hole of the coupler dimensioned to receive an end of the removable stirrer.

1 132. The apparatus of claim 131, further comprising a locking mechanism for  
2 preventing relative rotation of the coupler and the removable stirrer, the locking mechanism  
3 including:

4 a pin embedded in the end of the removable stirrer;  
5 a spring mounted between the coupler and a shoulder formed on the removable stirrer  
6 periphery; and

7 an axial groove extending from an entrance of the second hole to a lateral slot cut  
8 through a wall of the coupler, the lateral slot extending partway around the coupler  
9 circumference to an axial slot cut through the wall of the coupler;

10 wherein the axial groove, the lateral slot, and the axial slot are sized to accommodate  
11 the pin when the end of the removable stirrer is inserted into the second hole and rotated, and  
12 the pin is held in the axial slot by a force exerted by the spring.

1 133. The apparatus of claim 128, wherein the removable stirrer is snapped into the  
2 upper spindle portion.

1 134. A method of making and characterizing materials comprising:  
2 providing vessels with starting materials to form reaction mixtures;  
3 injecting a fluid into at least one of the vessels;  
4 stirring the reaction mixtures; and  
5 evaluating the reaction mixtures by measuring at least one characteristic of the  
6 reaction mixtures

1 135. The method of claim 134, wherein the fluid comprises a catalyst.

1 136. The method of claim 134, wherein the fluid comprises a catalyst poison.

1 137. The method of claim 134, wherein the fluid comprises a comonomer.

1 138. A method for monitoring parallel, combinatorial chemical reactions, the  
2 method comprising:

3 (a) communicating a plurality of measured values to a microprocessor, the plurality of  
4 measured values being associated, respectively, with the contents of a plurality of reactors  
5 during simultaneous reaction of two or more reactants in the plurality of reactors, the  
6 reactants or reaction environments varying between the plurality of reactors,

7 (b) displaying the measured values, and

8 (c) repeating steps (a) and (b) at least once during the reactions.

1 139. The method of claim 138 wherein the plurality of measured values are  
2 associated, respectively, with the reaction environments for the plurality of reactors.

3 140. The method of claim 138, wherein step (c) is performed at a predetermined  
4 sampling rate.

5 141. The method of claim 138, further comprising:  
6 changing a reaction parameter associated with one of the plurality of reactor vessels in  
7 response to the measured value to maintain the reactor vessel at a predetermined set point.

1 142. The method of claim 141, wherein the reaction parameter comprises a reactor  
2 vessel temperature.

1 143. The method of claim 141, wherein the reaction parameter comprises a reactor  
2 vessel pressure.

1 144. The method of claim 141, wherein the reaction parameter comprises a reactor  
2 vessel motor speed.

1 145. The method of claim 138, further comprising:

2 quenching a catalyst in one of the plurality of reactor vessels in response to the

3 measured value associated with the contents of the reactor vessel.

1 146. The method of claim 138 further comprising:  
2 calculating an experimental value for one or more of the plurality of reactors based on  
3 one or more measured values for that one or more reactors.

1 147. The method of claim 146, wherein the experimental value comprises  
2 temperature change.

1 148. The method of claim 146, wherein the experimental value comprises pressure  
2 change.

1 149. The method of claim 146, wherein the experimental value comprises percent  
2 conversion of starting material.

1 150. The method of claim 146, wherein the experimental value comprises viscosity.

1 151. The method of claim 146, further comprising displaying the experimental  
2 value.

1 152. A method for controlling parallel, combinatorial chemical reactions, the  
2 method comprising  
3 communicating a plurality of experimental values to a microprocessor, the plurality of  
4 experimental values being associated, respectively, with a property of reaction environments  
5 for a plurality of reactors during simultaneous reaction of two or more reactants in the  
6 plurality of reactors, the reactants or reaction environments varying between the plurality of  
7 reactors,  
8 comparing the plurality of experimental values, respectively, to a plurality of setpoints  
9 for the property, and  
10 changing the reaction environment in one or more of the reactors in response to a  
11 difference between the experimental value and the setpoint associated with that one or more  
12 reactors.

1 153. The method of claim 152, wherein changing the reaction environment in one  
2 or more of the plurality of vessels comprises terminating a reaction occurring in one or more

3 vessels, the set point comprises a conversion target, and the change in one or more of the set  
4 of experimental values comprises a change in percent conversion of starting material.

1 154. The method of claim 152 wherein displaying the set of experimental values  
2 comprises displaying a graphical representation of the set of experimental values.

1 155. The method of claim 154 wherein the graphical representation comprises a  
2 histogram.

1 156. A computer program on a computer-readable medium for monitoring parallel,  
2 combinatorial chemical reactions, the computer program comprising instructions to:

3 (a) receive a plurality of measured values, the plurality of measured values being  
4 associated, respectively, with the contents of a plurality of reactors during simultaneous  
5 reactions of two or more reactants, the reactants or reaction environments varying between  
6 the plurality of reactors,

7 (b) display the measured values, and

8 (c) repeating steps (a) and (b) at least once during the reactions.

1 157. The computer program of claim 156, further comprising instructions to:  
2 change a reaction parameter associated with one of the plurality of reactor vessels in  
3 response to the measured value.

4 158. A reactor control system for monitoring and controlling parallel, combinatorial  
5 chemical reactions, the reactor control system comprising:

6 a system control module for providing control signals to an integrated parallel reactor  
7 system, the control signals being based on a set of reaction parameters, the integrated parallel  
8 control system comprising a plurality of reactors and a monitoring and control system  
9 selected from the group consisting of a mixing monitoring and control system, a temperature  
10 monitoring and control system and a pressure monitoring and control system,

11 a data analysis module for receiving a plurality of measured values from the integrated  
12 parallel reactor system, directly or indirectly via the system control module, and for  
calculating a plurality of calculated values from the respective measured values, and

a user interface module for receiving the set of reaction parameters, and for displaying  
the plurality of measured values and the plurality of calculated values.

1 159. An apparatus for parallel processing of reaction mixtures comprising:  
2 vessels for containing the reaction mixtures;  
3 a stirring system for agitating the reaction mixtures; and  
4 a pressure control system that is adapted to maintain a first group of the vessels at a  
5 different pressure than a second group of the vessels during processing.

1 160. An apparatus for parallel processing of reaction mixtures comprising:  
2 vessels for containing the reaction mixtures;  
3 a pressure control system that is adapted to maintain the vessels at a pressure greater  
4 than about 10 psig; and  
5 a temperature control system that is adapted to maintain a first group of the vessels at  
6 a different temperature than a second group of the vessels.

1 161. An apparatus for parallel processing of reaction mixtures comprising:  
2 vessels for containing the reaction mixtures; and  
3 an injection system for introducing a fluid into the vessels at a pressure that is greater  
4 than about 10 psig.

1 162. A method of making and characterizing materials comprising:  
2 providing vessels with starting materials to form reaction mixtures;  
3 allowing the reaction mixtures in the vessels to react while stirring each of the  
4 reaction mixtures;  
5 evaluating the reaction mixtures by measuring at least one characteristic of the  
6 reaction mixtures during at least a portion of the reaction;  
7 wherein the reaction is carried out at about the same time for each of the reaction  
8 mixtures.

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